ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS by EPA 3050B Rev. 2 (1996)

Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
Were reagents of sufficient purity to permit accuracy of determinations?	5.1				
Were method and reagent blanks less than MDLs?	5.1				
Was reagent water interference free?	5.2				
Were samples collected in plastic or glass containers?	6.2				
Were nonaqueous samples refrigerated?	6.3				
Were samples mixed to homogeneity and sieved?	7.1				
Were sample aliquots weighed to the nearest 0.01 g?	7.1				
Were samples prepared for either GFAA **and** ICP-MS **or** FLAA **and** ICP-AES?	1.1				
Were method blanks carried through all steps with each preparation batch?	8.2				
Were spiked duplicate samples analyzed at a rate of 5%?	8.3				
GFAA and ICP-MS					
Digestion Block or Bath					
Was 10 mL of 1+1 HNO_3 added and sample aliquots mixed?	7.2				
Were samples covered and refluxed at 95°C±5°C for 10 to 15 minutes without boiling?	7.2				
Were samples then cooled and have 5 mL concentrated HNO_3 added to them?	7.2				
Were samples covered and refluxed for 30 minutes?	7.2				
If brown fumes were observed, was additional 5 mL of concentrated HNO_3 added and 30 minute reflux repeated until no brown fumes were observed?	7.2				
Notes/Comments:					

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Were samples refluxed at 95°C±5°C for two hours or until approximately 5 mL without boiling?	7.2				
After digestion, did samples have 30% H_2O_2 and water added to them?	7.2.1				
Was 30% H_2O_2 added to samples while warming them until effervescence was minimal or sample appearances remained unchanged?	7.2.2				
Was not more than 10 mL of 30% H_2O_2 added to samples?	7.2.2				
Were samples next digested at 95°C±5°C for two hours or until approximately 5 mL without boiling?	7.2.3				
After cooling, were samples diluted with water and filtered, centrifuged, or settled prior to analysis?	7.2.4				
Direct Energy Devices such as Microwaves					
Was 10 mL of 1+1 HNO_3 added and sample aliquots mixed?	7.2				
Were digestion vessels covered by a vapor recovery device?	7.2				
Were samples refluxed at 95°C±5°C for 5 minutes without boiling?	7.2				
Were samples then cooled and have 5 mL concentrated HNO_3 added to them?	7.2				
Were samples covered and refluxed for 5 mL minutes?	7.2				
If brown fumes were observed, was additional 5 mL of concentrated HNO_3 added and 5 minute reflux repeated until no brown fumes were observed?	7.2				
Were samples finally refluxed at 95°C±5°C for 10 minutes without boiling using a vapor recovery device?	7.2				
After digestion, did samples have 30% H_2O_2 and water added to them?	7.2.1				
Was 30% H_2O_2 added to samples while warming them until effervescence was minimal or sample appearances remained unchanged?	7.2.2				
Notes/Comments:					

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Was not more than 10 mL of 30% H_2O_2 added to samples?	7.2.2				
Were samples next heated to 95°C±5°C for 6 minutes without boiling to remain at 95°C±5°C for 10 minutes?	7.2.3				
After cooling, were samples diluted with water and filtered, centrifuged, or settled prior to analysis?	7.2.4				
FLAA and ICP-AES					
Was 10 mL of 1+1 HNO_3 added and sample aliquots mixed?	7.2				
Were digestion vessels covered by a vapor recovery device?	7.2				
Were samples refluxed at 95°C±5°C for 5 minutes without boiling?	7.2				
Were samples then cooled and have 5 mL concentrated HNO_3 added to them?	7.2				
Were samples covered and refluxed for 5 mL minutes?	7.2				
If brown fumes were observed, was additional 5 mL of concentrated HNO_3 added and 5 minute reflux repeated until no brown fumes were observed?	7.2				
Were samples finally refluxed at 95°C±5°C for 10 minutes without boiling using a vapor recovery device?	7.2				
After digestion, did samples have 30% H_2O_2 and water added to them?	7.2.1				
Was 30% H_2O_2 added to samples while warming them until effervescence was minimal or sample appearances remained unchanged?	7.2.2				

Notes/Comments:

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Was not more than 10 mL of 30% H_2O_2 added to samples?	7.2.2				
Were samples next heated to 95°C±5°C for 6 minutes without boiling to remain at 95°C±5°C for 10 minutes?	7.2.3				
Was 10 mL of concentrated HCL added to the digestates from above steps?	7.3				
For block/bath digestion, were samples next covered and either relfluxed for 15 minutes at 95°C±5°C?	7.3				
For microwave/direct heating, was 5 mL concentrated HCl and 10 mL H_2O_2 added to samples, samples covered with a vapor recovery device, and samples refluxed for 5 minutes at 95°C±5°C?	7.3				
Optional Steps to improve Solubilities/Recoveries of Antimony, Lead, Barium, and Silver					
Were 2.5 mL conc HNO_3 and 10 mL conc HCL added to weighed sample aliquots?	7.5				
Were samples next refluxed for 15 minutes? (no temp specified)	7.5				
Were samples then filtered through a Whatman No 41 filter papers and filtrate collected?	7.5.1				
Were filters rinsed with no more than 5 mL of hot (about 95°C) HCl and then 20 mL hot reagent water with rinsings collected with filtrates?	7.5.1				
Were residues and filter papers combined with 5 mL conc HCl and digested at 95°C±5°C until filter papers dissolved?	7.5.2				
Were the above digestates filtered with reagent water and collected into the other filtrate vessels?	7.5.2				
If precipitates formed in filtrates, was up to 10 mL of additional HCl added to dissolve precipitates?	7.5.3				
Notes/Comments:					